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The Operating experience of Nitrophosphate Plant

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Abstract

Pak Arab fertilizers private Limited has been operating its nitrophosphate plant based on the ODDA process since 1978. The technology has been supplied by Stamicarbon Holland and engineering by Udhe Germany. Nitrophosphate route provides an interesting avenue for producing an agronomically balanced complex fertilizer.

Numbers of improvements are made in original design since after installation to meet the performance guarantees. Rock Phosphate is the main raw material for NP production. Plant was designed on Jordan rock and later Morocco, Egyptian and Algerian rock phosphates and their blends are tested. The other bottle neck was CN separation centrifuges. Failure of this equipment kept the plant operation on low rate hence these were replaced with Filter drums a slow moving unit. This helped to improved production quality and service period. To improve environment, NOx abatement Urea solution is added to Dissolving reactor. Two new reactors added to system to control reaction rate and anti foaming consumption was reduced drastically and it also helps in increasing production capacity of plant.

The main thrust of this paper is to present the operating experience of nitrophosphate plant, improvements made at this complex to improve reliability of complex.

Keywords: Type your 3-5 keywords here, separated by semicolons, using American spelling and avoiding general and plural terms and multiple concepts (avoid, for example, 'and', 'of'). These keywords will be used for indexing purposes.

Nomenclature

MTPD	metric tonne per day
NP	Nitrophosphate
PFL	Pak Arab Fertilizers
CAN	Calcium Ammonium nitrate
P_2O_5	Phosphorous pentoxide
^0C	degree centigrade

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1. Introduction

Pakarab Fertilizers is one of the largest fertilizer complex in Pakistan and is the only fertilizer manufacturing facility producing compound fertilizer in the country. The company was established as a result of memorandum of understanding concluded between West Pakistan Industrial Development Corporation of Pakistan and Abu Dhabi National Oil Company on March 7, 1973 and a participation Agreement emerged on November 01, 1973 to establish a joint venture for the expansion and modernization of the company predecessor - Natural Gas Fertilizer Factory, Multan. Thus the Company was incorporated on November 12, 1973 with total authorized capital of Rs. 1000 Million.

Later West Pakistan Industrial Development Corporation of Pakistan share in the Company was assumed by National Fertilizer Corporation of Pakistan as a result of reformation of public sector corporations and Abu Dhabi National Oil Company share in the Company had been assumed by International Petroleum Investment Company, Abu Dhabi. On July 14, 2005, Pak Arab Fertilizers was privatized at a cost of 14.125 billion Pakistani Rupees by Reliance Export under the umbrella of Fatima group and Arif Habib group.

Pak Arab Fertilizers complex operates with 960 MTPD ammonia, 1380 MTPD Nitric Acid, 280 MTPD Urea, 1500 MTPD Calcium ammonium nitrate, 1015 MTPD Nitrophosphate and 192 MTPD of Carbon di oxide.

2. Nitrophosphate Complex

The Nitrophosphate complex of PFL operates on basic ODDA process licensed by Stamicarbon, Holland and detailed engineering by Udhe, Germany. NP plant was designed to get fertilizers product specification of Nitrogen 22.6 % and P_2O_5 22.6 %. At the time of start up product specification was not achieved so different modifications were made. Later on, more modifications were done to achieve the desired product quality. In 1999, product specifications were revised to Nitrogen 22.5 ± 0.5 % and P_2O_5 20.0 ± 0.5 % due to

The Main Steps of NP process is as under

- Rock Phosphate Storage and Handling
- Dissolution of rock phosphate with nitric acid
- Crystallization of Dissolving solution and separation of calcium nitrate crystals from nitrophosphate acid
- Ammoniation (i.e. neutralization of nitrophosphate acid)
- Prilling of the slurry and conditioning of the product

Calcium Nitrate is produced as a by product in the process and sent to CAN plant where it is used for the production of calcium ammonium nitrate fertilizer. Ammonium nitrate solution produced from CAN plant is used at NP plant for N_2:P_2O_5 ratio correction.

Rock phosphate is weighed and added into the first dissolving reactor, fitted with an agitator, where it is mixed with the proper amount of Nitric acid plus wash acid. The exothermic dissolution reactions raise the temperature of the mixture to about 60^0C. In the second stage dissolving reactor (also fitted with an agitator), the reaction is completed and the dissolved liquid flows over to the buffer tank via inerts removal settling tanks. Urea solution is also added in first dissolving reactor to control NO_x in off –gases.

In order to avoid foaming during the reactions, required quantities of antifoaming agent is sent to the first dissolving reactor.

The phosphate solution is cooled to about 40^0C by means of cooling water. Solution is then pumped to over head tank, and then solution is fed to the crystallization lines A, B, C, D, E and F. In the crystallizers calcium nitrate is crystallized out as calcium nitrate tetra hydrate crystals and separated in Rotary drum vacuum filter. The mother liquor from filter drum is pumped to the neutralizers. To obtain the proper NP ratio in the end product, a certain amount of ammonium nitrate solution from the calcium nitrate conversion plant is recycled to this tank. This solution must be neutralized before evaporation. The filtrate is pumped to the neutralizer. The neutralizer is also fed with a proper amount of ammonia in order to effect adequate neutralization. Being exothermic, the neutralization reaction will cause the temperature to rise up to 118^0C. The amount of ammonia is controlled by an automatic pH control which should be 3.5 ~ 4.5. Gases leaving the neutralizer are washed in scrubber. After neutralization, the NP solution must be concentrated. Evaporation is effectuated in circulation-type evaporators. The NP solution flows over from the neutralizer to evaporators; gases leaving the separators are condensed in mixing condensers. In this evaporation stage, the solution is concentrated from about 75 % to 99.5 %. The high concentrated solution flows over into a melt tank. From the melt tank the NP solution is pumped to the top of the prilling tower. The concentrated NP melt is fed to the NP tank located on the prilling tower. From this tank, the NP solution is fed to prilling bucket distributing the product in the form of droplets over the cross section of the prilling tower, in which

a rising air-flow is maintained by fans installed in the top part of the tower. During their fall the droplets solidify to prills. The prills are removed from the bottom of the tower by means of a scraper at about 95^0C. Prills are conveyed by means of a belt conveyor to a cooling unit, cooling is done by air induced by a blower. Cooled Product is sent to Handling plant for Bagging and storage. The schematic diagram of NP process highlighting all the sections of ODDA process is shown in figure 1.

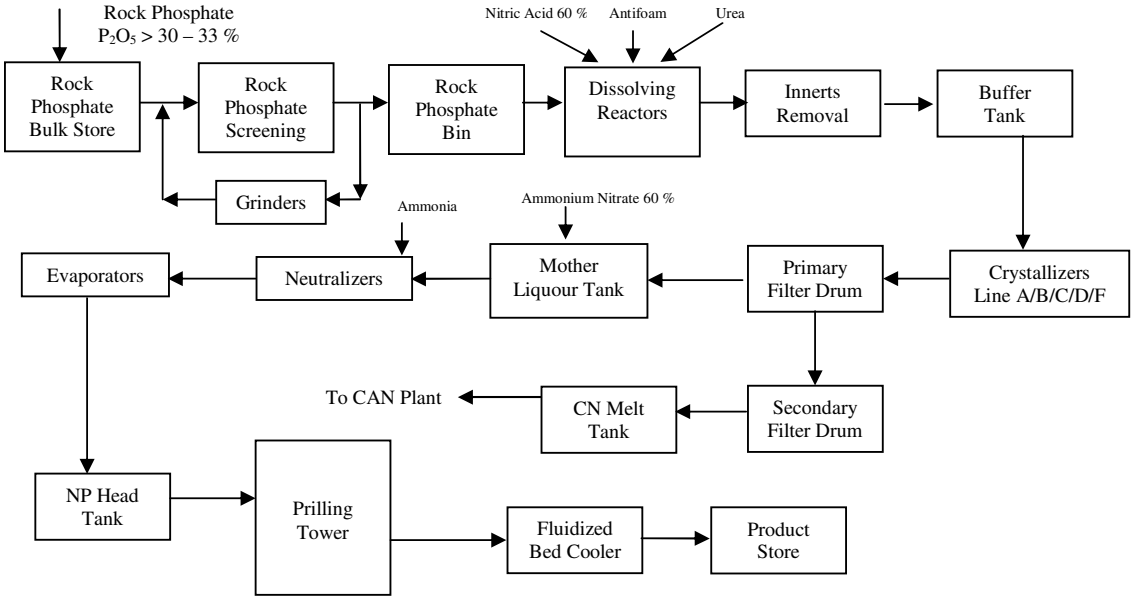

Fig. 1. Block diagram Nitrophosphate Plant

In order to keep plant in smooth operation a lot of in house modifications were carried out for debottlenecking of problems some of which are discussed in this paper

2.1 Trial of Rock phosphates of different Origins

NP plant was designed on Jordan Rock Phosphate but then due to high cost and availability issues Morocco Rock Phosphate started at plant in the year 1984. Later on, Egyptian and Algerian Rocks trial run also conducted at plant. The role of main constituents of rock phosphate is discussed below

a) P_2O_5-Phosphorous Pentoxide

The most important constituent of rock phosphate is P_2O_5 as this is the element required for fertilizer production. The ODDA section of our nitrophosphate plant is designed for a minimum P_2O_5 content of 33.4%. Hence any rock having P_2O_5 content lower than 33.4% is not suitable as it will increase the hydraulic load of the plant which has to be removed before granulation. Higher P_2O_5 rocks are beneficial as the impurities level in these rocks will be lower.

b) Silica

The silica content of the rock is in two forms. The active silica (clay) is beneficial as it reacts with fluorides in the rock and produces flousilicates which due to the low solubility, readily precipitates and finally reduces corrosion. The other silica (quartz) is inert which will not affect the process chemistry, but causes erosion in the various equipments of digester section. Hence the rock should have sufficient quantity of active silica to fix fluorides while the inert silica should be minimum. For this, it is recommended to have active silica/fluoride ratio of 0.5 and the total silica should not exceed 3%.

c) MgO (Magnesium oxide)

Acid soluble magnesium seems to be the main rock impurity that causes an increase in viscosity during ammoniation of NP melt. It should be as minimal as possible. MgO content above 0.3% in rock is detrimental for the process.

d) Al_2O_3/Fe_2O_3 (aluminium/iron oxide)

A certain amount of these oxides are required for achieving good calcium nitrate crystal growth and favorable lime size for filtration. However, higher amount of R_2O_3 causes very coarse lime which is not suitable for CAN plant and it also increases viscosity of the nitrophosphate melt in ANP plant causing load reduction and small reduction of water solubility of P_2O_5. R_2O_3 content of 0.4% ~ 0.8% is most favorable for ideal crystal growth of calcium nitrate as well as favorable lime size distribution.

2.1.1 Methodology for rock phosphate processing and evaluation

For any rock phosphate, about 10 - 12 standard parameters are analyzed and based on the level of different impurities; a broad prediction of its behavior is made. The analysis is compared with standard specification sheet (Table 1) and only those rocks whose analysis is conforming to exactly as per our requirement are cleared for further trial.

Table 1. Phosphate Rock Analysis

Analysis	PFL spec	Morocco	Egyptian	Algerian
P_2O_5	34.35	32.03	30.59	28.95
Silica	1.97	1.1	6.12	1.6
MgO	0.2	0.4	0.4	0.8
Al_2O_3	0.25	0.3	1.31	0.3
Fe_2O_3	0.28	0.15	0.7	0.25

The actual trials in the plant are carried out in a phase-wise manner by blending of 10% of trial rock with the main rock. The plant parameters are monitored and adjusted and the blend ratio is increased in steps of 10% after minimum observation at least for one month. The changed parameters are recorded for future use. This practice is also followed internationally by phosphatic fertilizer producers, while changing over the source of rock. Many a times blending of rocks helps in maintaining other parameters and the blended rock is more suitable than the two original rocks, if used independently.

2.1.1 Morocco Rock Phosphate

- High P2O5 due to which low rock consumption / ton of NP production
- Low silica contents increases plant on- stream hours and less erosion
- Ideal particle size distribution
- No adverse affect observed while testing this rock phosphate

2.1.2 Egyptian Rock Phosphate

- Load reduction when 100 % Egyptian rock used due to its high bulk density
- High silica and fine contents reduce life of conveyor belts
- More time for inerts cleaning due to high silica contents

2.1.3 Algerian Rock Phosphate

- Due to low Al_2O_3/Fe_2O_3 ratio fine crystals of calcium nitrate crystals formation which causes blockage and load reduction of rotary drum vacuum filters
- Nutrients in product could not be achieved due to low P_2O_5
- High MgO causes problems in ammoniation of NP solution and problems in evaporation system

2.2 Improvements in Inert Separation Section

In the design, three inert decanter centrifuges were installed; they had very high mechanical failure rate and operating cost. Main problem was with Decanter screw which frequently got damage. Also, no standby decanter was available. So, in case of problem in any centrifuge plant load had to be reduced and plant down time increases. Production of plant greatly suffers. Lot of consumption of hydraulic oil due to very frequent leakages also increases the operating cost. In the year 1982 these decanters were replaced with the battery of settling tanks. Three settling tanks are installed which can be operated in parallel or series as per requirement. That has reduced a lot of maintenance and operational

2.3 Improvement in CN separation

In the design, CN centrifuges were used for separation of CN crystals from mother liquor solution. But, again they have very high failure rate due to which they have high maintenance and operational cost.

The common maintenance problems were damaging of the separation screens, damaging of solution pusher ring and oil leakages due to vibrations. These screens were replaced with slow moving rotary filter drums. Double filtration technique was adopted successfully in the year 1996 with Komline Sanderson filter drum of USA. Now, three filter drums with one standby installed that has not only reduced greatly the maintenance cost but also has controlled slippage of P_2O_5 with CN. P_2O_5 in CN is controlled < 0.5 %.

2.4 Installation of new refrigeration loop

Two additional compressors were installed in the year 1982 for cooling of Brine solution with two additional trains of crystallizers. Which has not only improved quality of product but also reduce dependency on CAN plant for Ammonia consumption. Molar ratio CaO/P_2O_5 decreases resulting increase in water solubility of NP product. Addition of two crystallizer trains has increased plant capacity.

2.5 Installation of separate discharge line for NP melt Pumps

In original plant design only one discharge line was available to pump NP melt from ground floor to top of prilling tower. So in case of any blockage of line plant has to be shut down. This problem was removed by installing additional separate discharge lines for NP melt pumps to prilling tower in the year 1996 that has increased plant reliability and smooth operation.

2.6 Installation of new exhaust blower

In the design, one exhaust blower was installed, which was observed insufficient for exhaust gases discharge. This problem was rectified by installing two additional blowers which have made working area comfortable by reducing NOx and other gases in the year 1988.

2.7 Installation of Agitators in Neutralizers

In the design, there were no agitators in Neutralizers so pH of solution was not controlled and high pH causes decrease in water solubility of product. So for proper mixing of Ammonia with Mother Liquor agitators were installed which enable us to control pH. Also, two stages Neutralization pilot test run conducted successfully and its detail study for implementation is in progress.

2.8 Addition of New train of Dissolving reactors

Initially one train with two dissolving reactors were installed. Two trains with two dissolving reactors each installed in 2004 for more residence time and better dissolution of Rock Phosphate in Nitric Acid. That has not only improved reaction efficiency but also has improved production of plant. It has also reduced drastically antifoam consumption in dissolving reactor. Overall production of plant increases from 900 MeT/day to 1100 MeT/day.

2.9 Introduction of Urea control system to control NO_x emissions

Urea solution addition started in the year 2005 in dissolving reactors to control NO_x emission in stack. Now, NO_x emission in stack < 10 ppm before urea addition it was 200 ~ 300 ppm.

Conclusions

The Nitrophosphate Complex at PFL is unique in the sense that ammonium nitrophosphate and calcium ammonium nitrate plants are integrated with each other. The quality of rock processed and all other parameters affects directly the slurry behavior in the NP plant as well as the quality of byproduct lime from the NP plant which is used as raw material for the CAN plant. The choice of rock phosphate and adjustment of parameters while processing it have to therefore delicately balance the requirements of both.

While the operation of the Nitrophosphate Complex is well established over the years, we have been also successful in blending other rocks in various proportions in a controlled and scientific manner. Efforts to establish plant operation will continue in the future to achieve the best techno-economic benefits.